Electron Microprobe Analysis

Electron microprobe analysis is used for qualitative and semiquantitative elemental surface analysis of solids for elements from boron to plutonium. Elemental and compositional mapping of sample areas with a spatial resolution of about 1 μm is possible with this technique.

Principle of Technique

An electron beam is focused to a small spot on the surface of the sample. X rays with energies characteristic of the elements present are generated, and then detected and resolved by an energy- or wavelength-dispersive spectrometer. The x-ray intensity is proportional to the concentration of the corresponding element.

Samples

Form. Solids and powders.

Size. 1 mg to several hundred grams or ~9 × 2.5 cm in area.

Preparation. Ideally, the surface of the sample is polished metallographically to a 1-μm finish but rough materials can also be examined.

Limitations

Electron microprobe analysis measures only elements with Z ≥5 (boron).

The limits of detection are approximately 100 ppm. They are inversely proportional to *Z* and dependent on the sample matrix.

Estimated Analysis Time

After preparation of the sample, several minutes to a few hours are needed to perform an analysis.

Capabilities of Related Techniques

Scanning electron microscopy (SEM)/microprobe analysis has better photographic imaging capability than electron microprobe analysis. However, SEM/microprobe analysis has lower elemental resolution and its low-*Z* element limit is 11 (sodium).

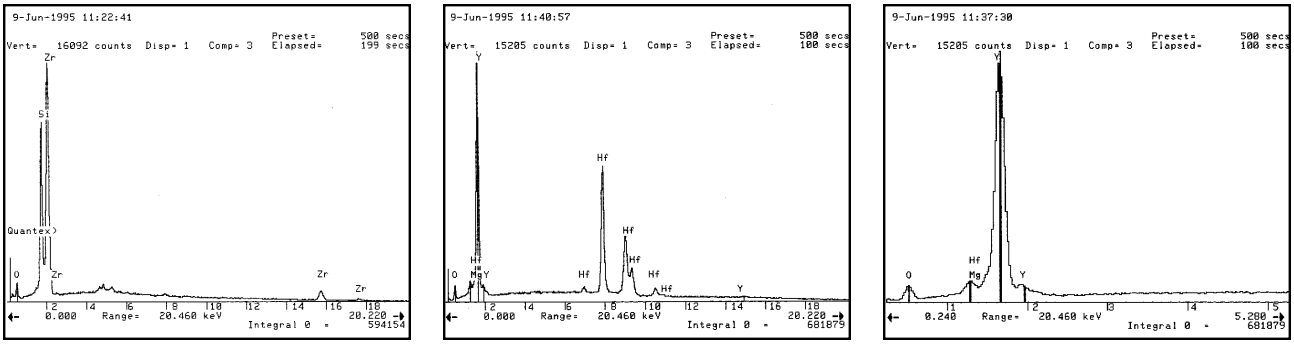
For bulk chemical analysis, x-ray fluorescence spectrometry is more accurate.

Analysis by the Materials Science and Technology Division

In addition to the analysis capabilities operated by ASD within the Plutonium Facility, the Materials Science and Technology Division of C&MS has the capability to perform electron microprobe and SEM analysis of a variety of materials.

Examples of Applications

- Measurement of individual microstructural phases in alloys, metals, ceramics, polymers, and minerals.
- Examinations of interfaces of ceramic-metal bonds.
- Identification of contaminants on surfaces.
- Determination of compositional (microscale) homogeneity.
- Analysis of compositional gradients at boundaries.



EDS spectrum of Zirconium/Silicon Oxide material. Other impurities for determination

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